

1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 10

VEGETATION SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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G. C. RONAN, DIRECTOR
Laboratory Services Branch
Ministry of the Environment

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1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 10

VEGETATION SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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and J C HIPFNER (editors)

Inorganic Trace Contaminants Section
Laboratory Services Branch
Ministry of the Environment

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INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = \sqrt{[(\sum x^2 - (\sum x)^2)/n/(n-1)]} \dots\dots I$$

$$sd = \sqrt{(\sum d^2/2n)} \dots\dots II$$

where : x = the individual values; n = the number of events
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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10. Vegetation

10.1 Vegetation Samples

Vegetation samples are collected, dried, ground and stored at room temperature. Sample preparation varies with the parameter set, and each set requires its own QA procedures and reference materials. QA samples consist of composited vegetation or other reference materials.

Table 10.1 summarizes the parameters determined, the preparation methods used and the instrument type used for the analysis of vegetation samples.

TABLE 10.1

Parameter	Collection Device	Preparation	Analysis
Metals	Plastic or glass	Acid digest	AAS, ICP-AES
Mercury	Plastic or glass	Acid digest	Cold Vapour AAS
Hydride Metals	Plastic or glass	Acid digest	AAS
Fluoride	Plastic or glass	Acid digest	ISE
Total N&P	Plastic or glass	Acid digest	Colorimetry
Uranium	Plastic or glass	Acid digest	ICP-MS
Chlorine	Plastic or glass	Pellet	XRF
Sulfur	Plastic or glass	Pellet	XRF
Potassium	Plastic or glass	Pellet	XRF

10.2 Vegetation Quality Assurance

Sample duplicates are prepared by taking a second aliquot from the prepared sample.

Reagent blanks are analysed with each analytical run. There are sufficient variations in the digestion acid lots that only one lot should be used in any one analytical run.

Matrix matched between-run composite samples are prepared by collecting samples in a large container. New composites are collected as the first is depleted or as the stability period expires. These composites may be spiked as necessary to provide a measureable level of analyte.

Table 10.2 indicates the sample descriptors used in the QA summary data, the source and the parameters that they are used to control.

TABLE 10.2

Sample Designation	Type	Parameter
QCV85-1,QCVB-2	Composite in-house veg	Metals,
Redoak	Composite in-house red oak leaves	Fluoride
Grass	Composite in-house grass material	Fluoride
Silvermapl	Composite in-house silver maple leaves	Fluoride
CON A	Composite veg sample	XRF(Cl,S,K)
CON B	Composite veg sample	XRF(Cl,S,K)
Orch Leaves	NBS Orchard Leaves 1571	As,Sb,Se,N, P
veg Control	Composite veg sample	As,Se,Sb
soil Cont1,2	Composite soil samples	As,SE,Sb
Con 684	Composite soil sample	Hg
Conv1,2	Composite veg samples	Uranium

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:ALUMINUM TEST CODE:ALUT SAMPLE TYPE:TERREST. VEG
UNIT:Vegetation/Soil/Sediment SUPERVISOR:L. Pastorek

METHOD CODE:533BA0 (JY)
REVISION NO:Original DATE:1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- 90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO3. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H2O2 and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Al}$)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS(Remarks)

Calibration Range: 0 - 10 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit:.031 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.62 $\mu\text{g/g}$ to 1000 $\mu\text{g/g}$

Accuracy- EPA #3 : 102.5 %

Precision of Controls-

	A	B
mean	100 $\mu\text{g/g}$	
std. dev.	11 $\mu\text{g/g}$	
R.S.D.	11 %	

Precision of Duplicates-low range	mid range	high range
s.d.	5.8	30
mean	71	370
		770

W 5 $\mu\text{g/g}$

T 25 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($\times 1.2\sigma$) Rejection Limits ($\times 1.3\sigma$)

Control Lower limit 78 67

OCV85-1 Upper limit 120 130

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN VEGETATION

Operating Range = 0.6000to 1000.0 ug/g

IN - RUN DUPLICATES

Range	<0.6000	0.6000to200.00	200.00to500.0	500.00to1000.0	>1000.0
no.	5	71	14	22	17
s.w.		5.7450	29.9670	177.5370	
mean		69.8100	367.8300	766.1300	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	229	99.890	11.0900	11.10

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	214	3.974	11.407

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:ALUMINUM TEST CODE:ALUT SAMPLE TYPE:TERREST. VEG
UNIT:Vegetation/Soil/Sediment SUPERVISOR:L. Pastorek

METHOD CODE:533BA0 (AS)
REVISION NO:Original DATE:1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- 90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Al)

INSTRUMENTATION: For the analysis of Ca, Mg, Al, Ti and/or B only. Inductively coupled plasma emission spectrometer -Atom Scan 2400 with autosampler, computer, & PET microcomputer interface to LIS(Remarks)

Calibration Range: 0 - 50 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: 1.0 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 5 $\mu\text{g/g}$ to 1000 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	100 $\mu\text{g/g}$	
std. dev.	3.6 $\mu\text{g/g}$	
R.S.D.	3.4 %	

Precision of Duplicates-low range mid range high range

s.d. 8.5

mean 360

W 5 $\mu\text{g/g}$

T 25 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 93 89

QCV85-1 Upper limit 110 110

REMARKS: Can also analyze by Jobin-Yvon ICP-AES.

- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM-AS IN VEGETATION

Operating Range = 5.0000to 1000.0 ug/g

IN - RUN DUPLICATES

Range	<5.0000	5.0000to200.00	200.00to500.0	500.00to1000.0	>1000.0
no.	1	0	1	0	0
s.w.		0.0000	8.5000	0.0000	
mean		0.0000	362.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	3	103.090	3.5520	3.45

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BL	40	-1.320	0.1259

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ALUMINUM TEST CODE: ALUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AAS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Al}$)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 20 mg/l

Resolution: 0.01 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 0.5 mg/l

Instrument Detection Limit: 0.04 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.80 $\mu\text{g/g}$ to 400 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	87 $\mu\text{g/g}$	
std. dev.	5.1 $\mu\text{g/g}$	
R.S.D.	5.9 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	2.9	8.5	ND
mean	32	100	ND

W 5 $\mu\text{g/g}$

T 25 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($\times 1.2\sigma$) Rejection Limits ($\times 1.3\sigma$)

Control Lower limit 77 72

QCV85-1 Upper limit 97 100

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM-AA IN VEGETATION

Operating Range = 0.8000to 400.0 mg/L

IN - RUN DUPLICATES

Range	<0.8000	0.8000to80.00	80.00 to200.0	200.00to400.0	>400.0
no.	0	4	1	0	0
s.w.		2.8720	8.4850	0.0000	
mean		32.0000	104.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	6	87.330	5.1250	5.87

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
------------	-----	------	-----------

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Antimony
UNIT: Biomaterials

TEST CODE: SBUT SBWT SAMPLE TYPE: Vegetation
SUPERVISOR: R. S. Sadana

METHOD CODE: 510EF3
REVISION NO: Original
NATURE OF LAST REVISION:

TYPE: Semi-aut. hydr. gen - flameless AAS
DATE: January, 1983

SAMPLE HANDLING:

Quantity Required- Approximately 2 g
Container- Glass jar with bakelite screw cap
Preservative- None
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90

Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.

Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear. Cool, add 0.5 ml of distilled water, then 2 ml conc. HCl. Dilute to 15 ml with distilled water and mix.

Feed the prepared solutions to the automated system for the determination of antimony by the hydride-FAAS technique.

INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.

REPORTING RESULTS: Two dec. places for <10, 1 dec. <100, whole no. if >100

INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200 & AA-5, with chart recorder, peristaltic pump and autosampler.

Open ended heated quartz "T" cell (0.6x10cm); gas-liquid separator.

Calibration Range: 0 - 40 ng/ml (linear <20 ng/ml)

Resolution: 0.01 absorbance (unexpanded scale)

Sensitivity: 0.02 µg/ml reads 0.15 abs.

Instrument Detection Limit: 0.001 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.3 to 10 µg/g

Accuracy- 99% (NBS orchard leaves)

Precision of Controls-

	A	B
mean	2.86	
std. dev.	.48	.45
R.S.D.	16 %	.18
		37 %

Precision of Duplicates-low range	mid range	high range
s.d.	.78	.65
mean	3.06	6.59

W 0.2 µg/g

T 1.0 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ANTIMONY IN VEGETATION

Operating Range = 0.3000to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.3000	0.3000to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	6	14	5	6	6
s.w.		0.2003	0.7755	0.6487	
mean		0.8720	3.0580	6.5920	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	6	2.855	0.4465	15.64
veg contro	14	0.484	0.1798	37.15
soil cont1	34	0.554	0.4929	88.97
soil cont2	26	18.446	3.4589	18.75

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Arsenic
UNIT: Biomaterials

TEST CODE: ASUT ASWT SAMPLE TYPE: Vegetation
SUPERVISOR: R. S. Sadana

METHOD CODE: 510EF3
REVISION NO: Original
NATURE OF LAST REVISION:

TYPE: Semi-aut. hydr. gen - flameless AAS
DATE: January, 1983

SAMPLE HANDLING:

Quantity Required- Approximately 2 g
Container- Glass jar with bakelite screw cap
Preservative- None
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90

Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.

Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear. Cool, add 0.5 ml of distilled water, then 2 ml conc. HCl. Dilute to 15 ml with distilled water and mix.

Feed the prepared solutions to the automated system for the determination of arsenic by the hydride-FAAS technique.

INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.

REPORTING RESULTS: Two dec. places for <10, 1 dec. <100, whole no. if >100

INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200 & AA-5, with chart recorder, peristaltic pump and autosampler.

Open ended heated quartz "T" cell (0.6x10cm); gas-liquid separator

Calibration Range: 0 - 40 ng/ml (linear <20 ng/ml)

Resolution: 0.01 absorbance (unexpanded scale)

Sensitivity: 0.02 µg/ml reads 0.15 abs.

Instrument Detection Limit: 0.001 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.3 to 10 µg/g

Accuracy- 99% (NBS orchard leaves)

Precision of Controls-

	A	B
mean	9.3	9.2
std. dev.	1.05	1.40
R.S.D.	11 %	15 %

Precision of Duplicates-low range

s.d.	.11
mean	.92

W 0.2 µg/g

mid range

.34
3.42

T 1.0 µg/g

high range

.91
7.32

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN VEGETATION

Operating Range = 0.3000 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.3000	0.3000 to 2.00	2.00 to 5.00	5.00 to 10.0	>10.0
no.	20	34	21	22	30
s.w.		0.1188	0.3396	0.9135	
mean		0.9910	3.4160	7.3150	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	45	9.297	1.0512	11.31
veg contro	68	9.184	1.4021	15.27
soil cont1	120	5.627	0.8063	14.33
soil cont2	104	12.216	1.5785	12.92

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BARIUM TEST CODE: BAUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0

REVISION NO: Original

DATE: 1980

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g

Container- Glass jar (4 oz) with bakelite screw cap

Preservative- None

Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90

Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Ba}$)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00172 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.04 $\mu\text{g/g}$ to 100 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	10 $\mu\text{g/g}$	
std. dev.	0.72 $\mu\text{g/g}$	
R.S.D.	7.2 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	0.53	0.88	14
mean	9.5	33	74

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 8.6 7.8

QCV85-1 Upper limit 11 12

REMARKS: -" % extracted" determined using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

BARIUM

IN VEGETATION

Operating Range = 0.0400 to 100.0 ug/g

IN - RUN DUPLICATES

Range	<0.0400	0.0400 to 20.00	20.00 to 50.00	50.00 to 100.0	>100.0
no.	5	47	38	21	17
s.w.		0.5330	0.8840	14.2101	
mean		9.4602	32.6570	74.0952	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	229	9.999	0.7223	7.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	183	0.100	0.3560

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BERYLLIUM TEST CODE: BEUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0

REVISION NO: Original

DATE: 1980

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g

Container- Glass jar (4 oz) with bakelite screw cap

Preservative- None

Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90

Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Be)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .000107 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.002 $\mu\text{g/g}$ to 10 $\mu\text{g/g}$

Accuracy- EPA #3: 95 %

Precision of Controls-

		A	B
	mean	.50 $\mu\text{g/g}$	
	std. dev.	$\mu\text{g/g}$	
	R.S.D.	%	
Precision of Duplicates-low range		mid range	high range
	s.d.	IS	IS
	mean	IS	IS
W 0.05 $\mu\text{g/g}$		T 0.25 $\mu\text{g/g}$	

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit

OCV85-1 Upper limit

REMARKS:- % extracted using orchard leaves (NBS-1571).

- IS - Insufficient data due to extremely low levels in samples.

SUMMARY REPORT OF QUALITY CONTROL DATA

BERYLLIUM IN VEGETATION

Operating Range = 0.0020 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.0020	0.0020 to 2.00	2.00 to 5.00	5.00 to 10.0	>10.0
no.	126	2	0	0	0
s.w.		0.2040	0.0000	0.0000	
mean		0.4173	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1.	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	87	0.018	0.0100

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BORON TEST CODE: BBUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (JY)

REVISION NO: Original

DATE: 1980

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g

Container- Glass jar (4 oz) with bakelite screw cap

Preservative- None

Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90

Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g B}$)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .0550 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 1 $\mu\text{g/g}$ to 500 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	43 $\mu\text{g/g}$	
std. dev.	4.9 $\mu\text{g/g}$	
R.S.D.	11 %	

Precision of Duplicates-low range	mid range	high range
s.d.	1.3	4.7
mean	12	32
		66

W 1 $\mu\text{g/g}$

T 5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 33

28

QCV85-1 Upper limit 53

58

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

BORON- γ IN VEGETATION

Operating Range = 1.0000 to 500.0 ug/g

IN - RUN DUPLICATES

Range	<1.0000	1.0000 to 100.00	100.00 to 250.0	250.00 to 500.0	>500.0
no.	0	128	1	0	0
s.w.		3.4647	1.8459	0.0000	
mean		24.6115	123.6999	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	230	43.402	4.9355	11.37

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	198	1.116	2.0148

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: BORON TEST CODE: BBUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO3. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H2O2 and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ B)

INSTRUMENTATION: For the analysis of Ca, Mg, Al, Ti and/or B only. Inductively coupled plasma emission spectrometer -Atom Scan 2400 with autosampler, computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 10 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: 0.05 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 1 $\mu\text{g/g}$ to 500 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	45 $\mu\text{g/g}$	
std. dev.	5.3 $\mu\text{g/g}$	
R.S.D.	12 %	

Precision of Duplicates-low range	mid range	high range
s.d.	1.0	23
mean	6.6	170
		290

W 1 $\mu\text{g/g}$

T 5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($\pm 2\sigma$) Rejection Limits ($\pm 3\sigma$)

Control Lower limit 34 29

OCV85-1 Upper limit 56 61

REMARKS: Can also analyze by Jobin-Yvon ICP-AES.

- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

BORON-AS IN VEGETATION

Operating Range = 1.0000to 500.0 ug/g

IN - RUN DUPLICATES

Range	<1.0000	1.0000to100.00	100.00to250.0	250.00to500.0	>500.0
no.	0	2	1	2	0
s.w.		1.0245	23.0191	6.8217	
mean		6.5864	174.8129	287.3838	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	12	44.620	5.2804	11.83

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BL	540	0.078	0.0278

DATE 87/04/15

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CADMIUM TEST CODE: CDUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to < 1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Cd}$)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00159 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.03 $\mu\text{g/g}$ to 2.5 $\mu\text{g/g}$

Accuracy- EPA #3 : 93.3 %

Precision of Controls-

	A	B
mean	0.80	
std. dev.	0.064	
R.S.D.	8.1%	

Precision of Duplicates-low range	mid range	high range	
s.d.	0.086	0.067	0.057
mean	0.29	0.78	1.6

W 0.1 $\mu\text{g/g}$

T 0.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($\pm 2\sigma$) Rejection Limits ($\pm 3\sigma$)

Control Lower limit 0.67 0.61

QCV85-1 Upper limit 0.93 0.99

REMARKS: -% extracted using orchard leaves (NBS-1571)

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN VEGETATION

Operating Range = 0.0200 to 2.5 ug/g

IN - RUN DUPLICATES

Range	<0.0200	0.0200 to 0.50	0.50 to 1.25	1.25 to 2.5	>2.5
no.	24	54	35	10	6
s.w.		0.0872	0.0668	0.0571	
mean		0.2517	0.7819	1.5516	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	230	0.799	0.0644	8.06

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	102	0.056	0.1140

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CADMIUM TEST CODE: CDUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AAS)
REVISION NO: ^Ariginal DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO3. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H2O2 and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Cd)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 0.5 mg/l

Resolution: 0.001 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 1.5 mg/l

Instrument Detection Limit: 0.004 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.08 $\mu\text{g/g}$ to 10 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	0.90 $\mu\text{g/g}$	
std. dev.	.052 $\mu\text{g/g}$	
R.S.D.	5.8 %	

Precision of Duplicates-low range mid range high range

s.d.	0.039
mean	0.54

W .025 $\mu\text{g/g}$

T .125 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	0.80	0.74
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OCV85-1 Upper limit	1.0	1.1
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REMARKS:- % extracted using orchard leaves (NBS-1571).

- spurious high results are possible at low concentrations unless background correction used.

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM-AA IN VEGETATION

Operating Range = 0.0800to 10.0 mg/L

IN - RUN DUPLICATES

Range	<0.0800	0.0800to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	0	8	0	0	0
s.w.		0.0390	0.0000	0.0000	
mean		0.5350	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	19	0.900	0.0520	5.78

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CALCIUM TEST CODE: CAUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (JY)

REVISION NO: Original

DATE: 1980

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g

Container- Glass jar (4 oz) with bakelite screw cap

Preservative- None

Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Ca}$)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 1000 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: 0.1498 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 3.0 $\mu\text{g/g}$ to 25000 $\mu\text{g/g}$

Accuracy-

Precision of Controls-

		A		B	
Precision of Duplicates-low range	mean	12000	$\mu\text{g/g}$		
	std. dev.	580	$\mu\text{g/g}$		
	R.S.D.	4.8	%		
W 50 $\mu\text{g/g}$	s.d.	84		mid range	high range
	mean	3100		360	720
				9100.	16000
		T 250 $\mu\text{g/g}$			

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($\pm 2\sigma$) Rejection Limits ($\pm 3\sigma$)

Control Lower limit 11000

10000

OCV85-1 Upper limit 13000

14000

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM-jy IN VEGETATION

Operating Range = 3.0000to 25000. ug/g

IN - RUN DUPLICATES

Range	<3.0000	3.0000to5000.0	5000.0to12500	12500.to25000.	>25000.
no.	2	42	46	34	5
s.w.		84.0950	362.9670	718.2510	
mean		3071.590	9054.840	15796.71	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	228	12179.76	580.2010	4.76

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	190	9.540	24.990

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CALCIUM TEST CODE: CAUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO3. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H2O2 and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Ca)

INSTRUMENTATION: For the analysis of Ca, Mg, Al, Ti and/or B only. Inductively coupled plasma emission spectrometer -Atom Scan 2400 with autosampler, computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2000 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: 4.0 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 50 $\mu\text{g/g}$ to 25000 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	13000 $\mu\text{g/g}$	
std. dev.	830 $\mu\text{g/g}$	
R.S.D.	6.3 %	

Precision of Duplicates-low range		mid range	high range
s.d.	230	320	820
mean	3000	7100	14000

W 200 $\mu\text{g/g}$

T 1000 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	11000	10000
QCV85-1 Upper limit	15000	16000

REMARKS: Can also analyze by Jobin-Yvon ICP-AES.

- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM-AS IN VEGETATION

Operating Range = 50.000to 25000. ug/g

IN - RUN DUPLICATES

Range	<50.000	50.000to5000.0	5000.0to12500	12500.to25000.	>25000.
no.	0	6	17	7	0
s.w.		197.4580	377.2100	1052.800	
mean		3428.910	8881.300	17069.00	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	71	13207.15	829.6200	6.28

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BL	3640	-1.750	0.6296

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: CHROMIUM TEST CODE: CRUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are freeze-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 96
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Cr)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00473 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.1 $\mu\text{g/g}$ to 25 $\mu\text{g/g}$

Accuracy- EPA #3 : 97 %

Precision of Controls-

	A	B
mean	6.4 $\mu\text{g/g}$	
std. dev.	1.1 $\mu\text{g/g}$	
R.S.D.	16 %	

Precision of Duplicates-low range	mid range	high range
s.d.	0.42	0.40
mean	2.7	8.0

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	4.2	3.1
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QCV85-1 Upper limit	8.6	9.7
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REMARKS: -" % extracted" determined using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN VEGETATION

Operating Range = 0.1000to 25.0 ug/g

IN - RUN DUPLICATES

Range	<0.1000	0.1000to5.00	5.00 to12.50	12.50 to25.0	>25.0
no.	19	81	24	4	1
s.w.		0.4269	0.4022	0.4076	
mean		2.5479	8.0086	15.4386	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	226	6.389	1.0519	16.46

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	202	0.332	0.8686

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COBALT TEST CODE: COUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO3. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H2O2 and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Co}$)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .003055 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.1 $\mu\text{g/g}$ to 10 $\mu\text{g/g}$

Accuracy- EPA #3 : 97 %

Precision of Controls-

	A	B
mean	1.2 $\mu\text{g/g}$	
std. dev.	.094 $\mu\text{g/g}$	
R.S.D.	7.5 %	

Precision of Duplicates-low range	mid range	high range
s.d.	0.28	0.18
mean	1.3	3.0
		6.6

W 0.2 $\mu\text{g/g}$

T 1.0 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 1.0 0.92

OCV85-1 Upper limit 1.4 1.5

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT

IN VEGETATION

Operating Range = 0.1000to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.1000	0.1000to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	35	42	35	12	5
s.w.		0.2816	0.1839	0.4641	
mean		1.2693	2.9778	6.5514	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	228	1.245	0.0935	7.52

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	87	0.108	0.2656

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COBALT TEST CODE: COUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BAO (AAS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Co}$)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 5 mg/l

Resolution: 0.01 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 7.0 mg/l

Instrument Detection Limit: 0.04 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.80 $\mu\text{g/g}$ to 100 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d.

mean

W .2 $\mu\text{g/g}$

T 1.0 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit

QCV85-1 Upper limit

REMARKS:- % extracted using orchard leaves (NBS-1571).

- spurious high results are possible at low concentrations unless background correction is used.

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT-AA IN VEGETATION

Operating Range = 0.8000to 100.0 mg/L

IN - RUN DUPLICATES

Range	<0.8000	0.8000to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	1	1	0	0
s.w.		0.0780	0.7070	0.0000	
mean		0.8550	41.5000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	0	0.000	0.0000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COPPER TEST CODE: CUUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to < 1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-88
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ of Cu)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .0047 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.1 $\mu\text{g/g}$ to 70 $\mu\text{g/g}$

Accuracy- EPA #3 : 112 %

Precision of Controls-

	A	B
mean	13 $\mu\text{g/g}$	
std. dev.	0.8 $\mu\text{g/g}$	
R.S.D.	6.2 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	0.50	0.99	1.5
mean	7.6	21	50

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 11 11

QCV85-1 Upper limit 15 15

REMARKS: -% extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER

IN VEGETATION

Operating Range = 0.1000 to 70.0 ug/g

IN - RUN DUPLICATES

Range	<0.1000	0.1000 to 14.00	14.00 to 35.00	35.00 to 70.0	>70.0
no.	0	115	11	3	0
s.w.		0.5017	0.9895	1.5411	
mean		7.5907	20.8134	50.2417	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	230	12.833	0.7978	6.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	204	0.170	0.2674

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COPPER TEST CODE: CUUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AAS)

REVISION NO: Original

DATE: 1980

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g

Container- Glass jar (4 oz) with bakelite screw cap

Preservative- None

Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 88

Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be eliminated by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Cu)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 5 mg/l

Resolution: 0.01 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 4.0 mg/l

Instrument Detection Limit: 0.02 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.40 $\mu\text{g/g}$ to 100 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	13 $\mu\text{g/g}$	
std. dev.	0.54 $\mu\text{g/g}$	
R.S.D.	4.1 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	0.13	0.71	1.4
mean	8.9	26	52

W 1 $\mu\text{g/g}$

T 5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 12

11

OCV85-1 Upper limit 14

15

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER-AA IN VEGETATION

Operating Range = 0.4000 to 100.0 mg/L

IN - RUN DUPLICATES

Range	<0.4000	0.4000 to 20.00	20.00 to 50.00	50.00 to 100.0	>100.0
no.	0	7	1	1	1
s.w.		0.1340	0.7070	1.4140	
mean		8.8790	26.5000	52.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	19	13.210	0.5350	4.05

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: IRON TEST CODE: FEUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-70
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Fe)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 50 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00209 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.04 $\mu\text{g/g}$ to 1000 $\mu\text{g/g}$

Accuracy- EPA #3 : 96 %

Precision of Controls-

	A	B
mean	230 $\mu\text{g/g}$	
std. dev.	18 $\mu\text{g/g}$	
R.S.D.	7.9 %	

Precision of Duplicates-low range	mid range	high range
s.d. 5.2	14	140
mean 93	310	760

W 5 $\mu\text{g/g}$

T 25 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 200 180

QCV85-1 Upper limit 270 280

REMARKS: -" % extracted" determined using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON

IN VEGETATION

Operating Range = 0.0400 to 1000.0 ug/g

IN - RUN DUPLICATES

Range	<0.0400	0.0400 to 200.00	200.00 to 500.0	500.00 to 1000.0	>1000.0
no.	7	57	22	26	17
s.w.		5.1610	13.7090	145.4890	
mean		93.1100	311.4200	759.4300	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	229	230.860	18.2140	7.89

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	204	2.200	4.0900

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: IRON TEST CODE: FEUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AAS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 70
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Fe)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 20 mg/l

Resolution: 0.01 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 5 mg/l

Instrument Detection Limit: 0.05 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.5 $\mu\text{g/g}$ to 400 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	270 $\mu\text{g/g}$	
std. dev.	26 $\mu\text{g/g}$	
R.S.D.	9.8 %	

Precision of Duplicates-low range	mid range	high range
s.d.		
mean		

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 220

190

OCV85-1 Upper limit 320

350

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON-AA IN VEGETATION

Operating Range = 0.5000 to 400.0 mg/L

IN - RUN DUPLICATES

Range	<0.5000	0.5000 to 80.00	80.00 to 200.0	200.00 to 400.0	>400.0
no.	0	1	0	0	0
s.w.		0.7070	0.0000	0.0000	
mean		65.5000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	4	267.500	26.3000	9.83

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: LEAD TEST CODE: PBUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-94
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO3. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H2O2 and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program. High sulphates in solution interfere chemically.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Pb)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .0314 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- .5 $\mu\text{g/g}$ to 50 $\mu\text{g/g}$

Accuracy- EPA #3 : 97 %

Precision of Controls-

	A	B
mean	19 $\mu\text{g/g}$	
std. dev.	2.0 $\mu\text{g/g}$	
R.S.D.	10 %	

Precision of Duplicates-low range	mid range	high range
s.d.	0.52	1.3
mean	3.1	19

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($\pm 2\sigma$) Rejection Limits ($\pm 3\sigma$)

Control Lower limit 15 13

QCV85-1 Upper limit 23 25

REMARKS: -" % extracted" determined using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN VEGETATION

Operating Range = 0.5000to 50.0 ug/g

IN - RUN DUPLICATES

Range	<0.5000	0.5000to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	28	60	21	20	0
s.w.		0.5590	1.3361	1.3516	
mean		3.0098	18.8142	36.0642	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	229	19.309	2.0160	10.44

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	169	0.784	1.3230

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: LEAD TEST CODE: PBUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AAS)

REVISION NO: Original

DATE: 1980

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g

Container- Glass jar (4 oz) with bakelite screw cap

Preservative- None

Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 94

Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Pb)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 20 mg/l

Resolution: 0.01 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 20 mg/l

Instrument Detection Limit: 0.06 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.5 $\mu\text{g/g}$ to 400 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	22 $\mu\text{g/g}$	
std. dev.	1.3 $\mu\text{g/g}$	
R.S.D.	5.9 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	.73	3.7	ND
mean	22.2	127	ND

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 19 18

OCV85-1 Upper limit 25 26

REMARKS:- % extracted using orchard leaves (NBS-1571).

- background correction must be used.

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD-AA IN VEGETATION

Operating Range = 0.5000 to 400.0 ug/g

IN - RUN DUPLICATES

Range	<0.5000	0.5000 to 80.00	80.00 to 200.0	200.00 to 400.0	>400.0
no.	0	9	6	0	0
s.w.		0.7300	3.7080	0.0000	
mean		22.2100	126.9200	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcv85-1	32	22.125	1.3137	5.94

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MAGNESIUM TEST CODE: MGUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (JY)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 92
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Mg)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 10 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: 0.203 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 4.1 $\mu\text{g/g}$ to 5000 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	2100 $\mu\text{g/g}$	
std. dev.	130 $\mu\text{g/g}$	
R.S.D.	6.0 %	

Precision of Duplicates-low range	mid range	high range
s.d.	140	96
mean	1500	3300

W 20 $\mu\text{g/g}$

T 100 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 1800

OCV85-1 Upper limit 2400

1700

2500

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM IN VEGETATION

Operating Range = 4.0000to 5000.0 ug/g

IN - RUN DUPLICATES

Range	<4.0000	4.0000to1000.0	1000.0to2500.	2500.0to5000.0	>5000.0
no.	1	54	54	18	2
s.w.		19.8380	139.5800	96.0890	
mean		642.9400	1501.120	3293.140	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	229	2099.700	125.6570	5.98

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	102	2.540	2.7860

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MAGNESIUM TEST CODE: MGUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 92
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Mg)

INSTRUMENTATION: For the analysis of Ca, Mg, Al, Ti and/or B only.

Inductively coupled plasma emission spectrometer -Atom Scan 2400 with autosampler, computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 50 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: 2.0 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 40 $\mu\text{g/g}$ to 5000 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	2300 $\mu\text{g/g}$	
std. dev.	63 $\mu\text{g/g}$	
R.S.D.	2.7 %	

Precision of Duplicates-low range	mid range	high range
s.d.	86	61
mean	1800	2641

W 50 $\mu\text{g/g}$

T 250 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	2200	2100
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OCV85-1 Upper limit	2400	2500
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REMARKS: Can also analyze by Jobin-Yvon ICP-AES.

- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM-AS IN VEGETATION

Operating Range = 40.000to 5000.0 ug/g

IN - RUN DUPLICATES

Range	<40.000	40.000to1000.0	1000.0to2500.	2500.0to5000.0	>5000.0
no.	0	0	1	1	0
s.w.		0.0000	86.3200	61.1000	
mean		0.0000	1847.100	2642.000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	7	2340.950	63.0350	2.69

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BL	40	8.840	0.6250

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MANGANESE TEST CODE: MNUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-91
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Mn)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00698 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.1 $\mu\text{g/g}$ to 250 $\mu\text{g/g}$

Accuracy- EPA #3 : 110 %

Precision of Controls-

	A	B
mean	60 $\mu\text{g/g}$	
std. dev.	4.6 $\mu\text{g/g}$	
R.S.D.	7.6 %	

Precision of Duplicates-low range	mid range	high range
s.d. 1.3	2.8	6.0
mean 29	80	170

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 51 46

QCV85-1 Upper limit 69 74

REMARKS: - " % extracted " determined using orchard leaves (NBS-1571).

- Low results may be due to poor ashing

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN VEGETATION

Operating Range = 0.1000 to 250.0 ug/g

IN - RUN DUPLICATES

Range	<0.1000	0.1000 to 50.00	50.00 to 125.0	125.00 to 250.0	>250.0
no.	7	27	23	23	49
s.w.		1.2644	2.7705	6.0445	
mean		29.0935	79.9945	167.1811	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	227	60.009	4.5704	7.62

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	130	0.244	0.5274

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MANGANESE TEST CODE: MNUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AAS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 91
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Mn)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 5 mg/l

Resolution: 0.01 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 2.5 mg/l

Instrument Detection Limit: 0.04 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.50 $\mu\text{g/g}$ to 100 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	270 $\mu\text{g/g}$	
std. dev.	8.2 $\mu\text{g/g}$	
R.S.D.	3.0 %	

Precision of Duplicates-low range	mid range	high range
s.d.	ND	ND
mean		

W .5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit

OCV85-1 Upper limit

REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE-AA IN VEGETATION

Operating Range = 0.5000to 100.0 mg/L

IN - RUN DUPLICATES

Range	<0.5000	0.5000to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	0	0	0	1
s.w.		0.0000	0.0000	0.0000	
mean		0.0000	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	4	270.000	8.1650	3.02

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Mercury
UNIT: Biomaterials

TEST CODE: HGUT HGWT SAMPLE TYPE: Vegetation
SUPERVISOR: R. S. Sadana

METHOD CODE: 541AF1
REVISION NO: Original
NATURE OF LAST REVISION:

TYPE: Flameless AAS
DATE: May, 1984

SAMPLE HANDLING:

Quantity Required- Approx. 1 g
Container- Glass vials or jars
Preservative- None
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Weigh approx. 0.250 g of sample into
a 50 ml Folin-Wu digestion tube. Add 5 ml of acid mixture
(4:1 -H₂SO₄:HNO₃) and place the tube in a Technicon aluminum hot block
(1½ h @ 150°C; 1½ h @ 190°C; 2 h @ 250°C).
Cool overnight, then dilute to 25 ml with distilled water.
Run in batches of about 28 samples.

Treat blanks and calibration standards in exactly the same manner.
Determine mercury in the entire volume. The measurement step is
automated and is based on the evolution of atomic vapour of mercury
(wavelength - 254nm) by the addition of a reducing agent.

INTERFERENCES: Water vapour; organic solvents.
Very high concentration of cations.

REPORTING RESULTS: Two significant figures (ug/g).

INSTRUMENTATION: Automated sampler and peristaltic pump
(Technicon or Gilson). Laboratory Data Control U.V. monitor
(Pharmacia or Milton-Roy)

Calibration Range: 0 - 20.0 ng/ml

Resolution: 0.4 ng/ml (one division on recorder chart paper)

Sensitivity: 1.0 ng/ml reads 0.05 absorbance (2.5 divs on chart)

Instrument Detection Limit: 0.1 ng/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 - 4.0 µg/g

Accuracy- 90% at 1.1 µg/g

Precision of Controls-

mean 0.454

std. dev. 0.035

R.S.D. 7.6 %

Precision of Duplicates-low range

s.d. .018

mean .454

W .01 µg/g

mid range

high range

.18

2.45

T .05 µg/g

CONTROL LIMITS:

REMARKS:

- Precision based on CCIW round robin.
- Detection Limit - 2x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MERCURY IN VEGETATION

Operating Range = 0.0100to 4.0 ug/g

IN - RUN DUPLICATES

Range	<0.0100	0.0100to0.80	0.80 to2.00	2.00 to4.0	>4.0
no.	2	18	0	1	1
s.w.		0.0183	0.0000	0.1768	
mean		0.1690	0.0000	2.4450	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
con 684	104	0.454	0.0347	7.64

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0	0

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MOLYBDENUM TEST CODE: MOUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Mo)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00422 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.08 $\mu\text{g/g}$ to 5.0 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	0.66 $\mu\text{g/g}$	
std. dev.	0.14 $\mu\text{g/g}$	
R.S.D.	21 %	

Precision of Duplicates-low range	mid range	high range
s.d.	0.29	0.11
mean	0.66	1.4

W 0.2 $\mu\text{g/g}$

T 1.0 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	0.38	0.24
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OCV85-1 Upper limit	0.94	1.1
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REMARKS: -" % extracted" determined using orchard leaves (NBS-1571).
-Spurious high results at low concs. unless background corr.

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN VEGETATION

Operating Range = 0.0800 to 5.0 ug/g

IN - RUN DUPLICATES

Range	<0.0800	0.0800 to 1.00	1.00 to 2.50	2.50 to 5.0	>5.0
no.	90	28	6	1	3
s.w.		0.3047	0.1076	0.2461	
mean		0.5192	1.3676	3.4141	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	125	0.660	0.1357	20.58

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	134	0.818	3.6322

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: NICKEL TEST CODE: NIUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 Oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to < 1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-85
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Ni)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00513 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.10 $\mu\text{g/g}$ to 50 $\mu\text{g/g}$

Accuracy- EPA #3 : 103 %

Precision of Controls-

	A	B
mean	12 $\mu\text{g/g}$	
std. dev.	0.64 $\mu\text{g/g}$	
R.S.D.	5.4 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	0.83	0.51	1.1
mean	4.1	16	39

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	10	10
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QCV85-1 Upper limit	14	14
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REMARKS: -% extracted using orchard leaves (NBS-1751).

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN VEGETATION

Operating Range = 0.1000 to 50.0 ug/g

IN - RUN DUPLICATES

Range	<0.1000	0.1000 to 10.00	10.00 to 25.00	25.00 to 50.0	>50.0
no.	17	78	18	8	8
s.w.		0.8214	0.5113	1.0570	
mean		3.9454	15.8383	38.6142	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	227	11.812	0.6415	5.43

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	190	0.198	0.4864

DATE 87/03/31

10.60

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: NICKEL TEST CODE: NIUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AAS)

REVISION NO: Original

DATE: ^A980

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g

Container- Glass jar (4 oz) with bakelite screw cap

Preservative- None

Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 85
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO3. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H2O2 and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Ni}$)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 5 mg/l

Resolution: 0.01 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 7.0 mg/l

Instrument Detection Limit: 0.04 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.80 $\mu\text{g/g}$ to 100 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	14 $\mu\text{g/g}$	
std. dev.	0.64 $\mu\text{g/g}$	
R.S.D.	4.6 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	0.42	$\mu\text{g/g}$	2.8
mean	2.90		94

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 13

12

QCV85-1 Upper limit 15

16

REMARKS:- % extracted using orchard leaves (NBS-1571).

- spurious high results are possible at low conc. unless background correction used.

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL-AA IN VEGETATION

Operating Range = 0.8000to 100.0 mg/L

IN - RUN DUPLICATES

Range	<0.8000	0.8000to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	1	0	1	1
s.w.		0.4240	0.0000	2.8280	
mean		2.9000	0.0000	94.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	8	13.880	0.6410	4.62

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Potassium TEST CODE: KKUT KKWT SAMPLE TYPE: Vegetation
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 009AX2 TYPE: X-ray fluorescence
REVISION NO: 84-01 DATE: August, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g.
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- Pelletized samples must be stored in dessicator
Other- Air dried, and ground to less than 1 mm in a Wiley Mill.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-

Procedure- Transfer 2.00 g sample to a plastic vial to which 0.50 g Hoeschst "C" wax is added.
Mix thoroughly and transfer to briquetting die. Press at 20 tons/sq. in. for 60 - 90 secs for smooth, durable, homog. pellet.
Analyze using a Siemens SRS-1 X-ray fluorescence spectrometer with following parameters: kV - 45; mA - 30; base line - 1.3; width - 2.5; collimator - 0.4°; crystal PET; angle - 117.660; detector - flow counter.
Intensity is determined from the average of two 4 sec. counts.
Concentration is determined from stored calibration data.

INTERFERENCES: Analysis protocol corrects for matrix effects due to Si, Cl, Ca, P, S.

REPORTING RESULTS: K as per cent to two decimal places

INSTRUMENTATION: Siemens SRS-1 X-ray fluorescence spectrometer with HP9825 and Commodore Pet computers; also briquetting die, Sartorius 4-place balance, Spex hydraulic press, Herzog hydraulic press.

Calibration Range: 0.37% to 4.46%

Resolution:

Sensitivity:

Instrument Detection Limit: 0.02%

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.01 to 2.0%

Accuracy- 98.8% at 1.56% level

Precision of Controls-

	A	B
mean	1.56 %	4.42 %
std. dev.	.017 %	.073 %
R.S.D.	1.1 %	1.7 %

Precision of Duplicates-low range	mid range	high range
s.d.	0.012	0.107
mean	0.21	0.70

W .01 %

T .05 %

CONTROL LIMITS:

REMARKS:

- Samples of 1 g can be prepared when there is insufficient material for a 2 g pellet.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

POTASSIUM IN VEGETATION

Operating Range = 0.02 to 2.0 %

IN - RUN DUPLICATES

Range	<0.02	0.02 to 0.40	0.40 to 1.00	1.00 to 2.0	>2.0
no.	0	66	56	61	35
S.W.		0.0120	0.1070	0.0310	
mean		0.1900	0.7000	1.4900	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CON A	60	1.560	0.0170	1.09
CON B	60	4.420	0.0730	1.65

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/09/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Selenium
UNIT: Biomaterials

TEST CODE: SEUT SEWT SAMPLE TYPE: Vegetation
SUPERVISOR: R. S. Sadana

METHOD CODE: 510EF3
REVISION NO: Original
NATURE OF LAST REVISION:

TYPE: Semi-aut. hydr. gen - flameless AAS
DATE: January, 1983

SAMPLE HANDLING:

Quantity Required- Approximately 2 g
Container- Glass jar with bakelite screw cap
Preservative- None
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Weigh 60 mg (45 mesh) sample into a 18 x 150 mm

pyrex graduated test tube. Add 3 ml of acid mixture
(6 nitric: 3 sulphuric: 1 perchloric). Process in batches
of 80 samples including blanks, calibration standards
and controls.

Digest in an aluminum hot block at a medium setting on
the hot plate for 14 hrs until dense white fumes appear.
Cool, add 0.5 ml of distilled water, then 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.

Feed the prepared solutions to the automated system for the
determination of selenium by the hydride-FAAS technique.

INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may
interfere.

REPORTING RESULTS: Two dec. places for <10, 1 dec. <100, whole no. if >100

INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron
1200 & AA-5, with chart recorder, peristaltic pump and autosampler.

Open ended heated quartz "T" cell (0.6x10cm); gas-liquid separator.

Calibration Range: 0 - 40 ng/ml (linear <20 ng/ml)

Resolution: 0.01 absorbance (unexpanded scale)

Sensitivity: 0.02 µg/ml reads 0.20 abs.

Instrument Detection Limit: 0.001 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.3 to 10 µg/g

Accuracy- 99% (NBS orchard leaves)

Precision of Controls-

mean .082

std. dev. .052

R.S.D. 64 %

B

3.74

.40

11 %

Precision of Duplicates-low range

s.d. .044

mean .523

mid range

.43

3.88

high range

ND

ND

W 0.2 µg/g

T 1.0 µg/g

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

SELENIUM IN VEGETATION

Operating Range = 0.3000 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.3000	0.3000 to 2.00	2.00 to 5.00	5.00 to 10.0	>10.0
no.	30	26	4	0	0
s.w.		0.0472	0.4272	0.0000	
mean		0.6970	3.8750	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
orch leave	31	0.082	0.0523	63.78
veg contro	49	3.741	0.4038	10.79
soil cont1	66	0.510	0.1506	29.53
soil cont2	68	0.699	0.2396	34.28

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/17

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: SILVER TEST CODE: AGUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 590AA1 (AAS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 1 ml H₂SO₄ and 2.5 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix well.
Analyze by atomic absorption spectrophotometry (AAS).

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Ag}$)

INSTRUMENTATION:

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 0.500 mg/l

Resolution: 0.001 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 2.5 mg/l

Instrument Detection Limit: 0.005 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.02 $\mu\text{g/g}$ to 10.0 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	0.23 $\mu\text{g/g}$	
std. dev.	.061 $\mu\text{g/g}$	
R.S.D.	26 %	

Precision of Duplicates-low range	mid range	high range
s.d.	0.024	
mean	0.212	

W 0.02 $\mu\text{g/g}$

T 0.10 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	0.11	0.047
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QCV85-1 Upper limit	0.35	0.41
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REMARKS:- % extracted -ND: determined using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

SILVER-AA IN VEGETATION

Operating Range = 0.0200 to 10.0 mg/L

IN - RUN DUPLICATES

Range	<0.0200	0.0200 to 2.00	2.00 to 5.00	5.00 to 10.0	>10.0
no.	0	6	0	0	1
s.w.		0.0240	0.0000	0.0000	
mean		0.2120	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	7	0.230	0.0610	26.52

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: SODIUM TEST CODE: NAUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA1 (AAS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic emission spectrophotometry (AES).

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Na}$)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 20 mg/l

Resolution: 0.001 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 0.5 mg/l

Instrument Detection Limit: 0.001 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0.02 $\mu\text{g/g}$ to 400 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	320 $\mu\text{g/g}$	
std. dev.	32 $\mu\text{g/g}$	
R.S.D.	10 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	2.8	8.2	31 $\mu\text{g/g}$
mean	24	120	310

W 2 $\mu\text{g/g}$

T 10 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)
Control Lower limit 270 220
QCV85-1 Upper limit 380 420

REMARKS:- % extracted -ND: determined using orchard leaves (NBS-1571).
- analyzed in emission mode.

SUMMARY REPORT OF QUALITY CONTROL DATA

SODIUM IN VEGETATION

Operating Range = 0.0200 to 400.0 ug/g

IN - RUN DUPLICATES

Range	<0.0200	0.0200 to 80.00	80.00 to 200.0	200.00 to 400.0	>400.0
no.	2	46	12	18	7
s.w.		2.5600	8.1700	30.6870	
mean		20.5200	124.5800	308.0600	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qcv85-1	165	317.394	32.3829	10.20

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/04/03

10.70

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: STRONTIUM TEST CODE: SRUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original
NATURE OF LAST REVISION:

DATE: 1980

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Sr)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00129 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.026 $\mu\text{g/g}$ to 200 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	34 $\mu\text{g/g}$	
std. dev.	1.7 $\mu\text{g/g}$	
R.S.D.	5.0 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	1.23	1.85	7.32
mean	16.9	57.6	115

W 0.2 $\mu\text{g/g}$

T 1.0 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($\pm 2\sigma$) Rejection Limits ($\pm 3\sigma$)

Control Lower limit 31

29

OCV85-1 Upper limit 37

39

REMARKS: -" % extracted" determined using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

STRONTIUM IN VEGETATION

Operating Range = 0.0300 to 200.0 ug/g

IN - RUN DUPLICATES

Range	<0.0300	0.0300 to 40.00	40.00 to 100.0	100.00 to 200.0	>200.0
no.	2	85	34	8	0
s.w.		1.2270	1.8537	7.3138	
mean		16.9865	57.6126	114.9634	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	228	33.953	1.6817	4.95

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	169	0.074	0.3332

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: TITANIUM TEST CODE: TIUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA1 (AS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-
Procedure-Weigh 0.250 g of sample into a calibrated pyrex test-tube. Muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with 1-2 drops DDW, followed by 0.25 ml H₂SO₄. Mix and stand at room temperature for 15 - 30 min. Place in aluminum heating strips and digest on a hot plate for 2 hrs or until almost dry. Samples MUST fume. Remove from the hot plate, allow to cool and add another 0.25 ml H₂SO₄. Repeat the digestion; when the samples have cooled add 2 drops of H₂O₂ and dilute to 5 ml with DDW. Mix the contents and analyze by Atom Scan 2400 (ICP-AES).

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g Ti}$)

INSTRUMENTATION:

Inductively coupled plasma emission spectrometer -Atom Scan 2400 with autosampler and computer. PET microcomputer interface to LIS

Calibration Range: 0 - 10 mg/l

Resolution: 0.0001 mg/l

Sensitivity: NA

Instrument Detection Limit: 0.05 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.10 $\mu\text{g/g}$ to 50 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	11 $\mu\text{g/g}$	78 $\mu\text{g/g}$
std. dev.	1.2 $\mu\text{g/g}$	5.7 $\mu\text{g/g}$
R.S.D.	11 %	7.3 %

Precision of Duplicates-	low range	mid range	high range
s.d.	0.22	ND	0.00
mean	1.9	ND	29

W 0.1 $\mu\text{g/g}$

T 0.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($\pm 2\sigma$) Rejection Limits ($\pm 3\sigma$)

Control Lower limit	8.6	67	7.4	61
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QCV85-1 Upper limit	13	89	15	95
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REMARKS: Control limits for A & B (Low & High, respectively)

- " % extracted" - ND. Determined using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

TITANIUM-AS IN VEGETATION

Operating Range = 0.1000 to 50.0 ug/g

IN - RUN DUPLICATES

Range	<0.1000	0.1000 to 10.00	10.00 to 25.00	25.00 to 50.0	>50.0
no.	0	3	0	1	0
s.w.		0.2236	0.0000	0.0000	
mean		1.9333	0.0000	29.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
v85-1	8	5.818	1.2317	21.17

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: VANADIUM TEST CODE: VVUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted->90
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO3. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H2O2 and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g V}$)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .00751 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.2 $\mu\text{g/g}$ to 10 $\mu\text{g/g}$

Accuracy- EPA #3: 96 %

Precision of Controls-

	A	B
mean	0.79 $\mu\text{g/g}$	
std. dev.	0.28 $\mu\text{g/g}$	
R.S.D.	35 %	

Precision of Duplicates-	low range	mid range	high range
s.d.	0.47	0.74	0.32
mean	1.4	3.2	6.2

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 0.23

OCV85-1 Upper limit 1.35

REMARKS: -" % extracted" determined using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM

IN VEGETATION

Operating Range = 0.2000 to 10.0 ug/g

IN - RUN DUPLICATES

Range	<0.2000	0.2000 to 2.00	2.00 to 5.00	5.00 to 10.0	>10.0
no.	62	28	23	16	0
s.w.		0.4746	0.7346	0.3155	
mean		0.9038	3.1860	6.2524	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	34	0.791	0.2785	35.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	107	0.186	0.1910

DATE 87/03/31

10.76

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: URANIUM TEST CODE: UUUT SAMPLE TYPE: VEGETATION
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 520AE2

REVISION NO: 2

DATE: Feb., 1986

NATURE OF LAST REVISION: Detectn meth & prepn. from fluoro to ICP/MS.

SAMPLE HANDLING:

Quantity Required- Approximately 20 g.

Container- Glass jars (4 oz) with bakelite/screw cap or PET container

Preservative- None

Other- Air-dried and ground to less than 1 mm in a Wiley Mill.

SAMPLE PREPARATION: Partial Extn.- Yes Total Extn.- % Extracted-

Procedure- Ash 0.2 g at 500C for 6 hrs in a crucible. Add 2 ml of 8N HNO₃ to cooled sample. Heat at a low heat until 1 ml of sample in solution is left.

Wash into 50 ml graduated centrifuge tube and make up to 50 ml.

Centrifuge and analyze supernatant by ICP/MS.

INTERFERENCES: High concentrations of alkali metals.

REPORTING RESULTS: ug/g U.

INSTRUMENTATION: Sciex Elan 250 inductively coupled plasma mass spectrometer.

Calibration Range: 0 to 1.0 mg/l.

Resolution: 0.0001 mg/l.

Sensitivity: 0.100 mg/l - between 35000 and 85000 counts per second

Instrument Detection Limit: 0.0001 mg/l.

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.005 to 20 µg/g.

Accuracy- Not determined (ND).

Precision of Controls-

	A	B
mean	.42 µg/g	11.1 µg/g
std. dev.	.071 µg/g	.78 µg/g
R.S.D.	16 %	7.0 %

Precision of Duplicates-	low range	mid range	high range
s.d.	0.047	0.176	.629
mean	0.298	7.050	15.74

W 0.2 µg/g

T 1.0 µg/g

CONTROL LIMITS: µg/g	W.L. (x ±2σ)	R.L. (x±3σ)
Control L.L.	9.5	8.7
U.L.	12.7	13.5

REMARKS:- Control limits:

- > ± 3 sd (standard deviations) on digested control samples before rejection of run.

- Conversion from mg/l to µg/g: µg/g = mg/l x 250.

SUMMARY REPORT OF QUALITY CONTROL DATA

URANIUM IN VEGETATION

Operating Range = 0.0050 to 20.0 ug/g

IN - RUN DUPLICATES

Range	<0.0050	0.0050 to 4.00	4.00 to 10.00	10.00 to 20.0	>20.0
no.	4	29	2	3	0
s.w.		0.0470	0.1758	0.6293	
mean		0.2980	7.0500	15.7390	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
conv1	21	0.423	0.0707	16.71
conv2	38	11.109	0.7816	7.04

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ZINC TEST CODE: ZNUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to < 1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted-96
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube and make up to 10 ml with DDW; mix well. Analyze by AES-ICP. Use the Jobin-Yvon I for samples requiring 5 or more elements and the Atom Scan 2400 for those requiring only Ca, Mg, Al, Ti and/or B.

INTERFERENCES: Several, which are compensated for by the computer program.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Zn)

INSTRUMENTATION: For the analysis of 5 or more elements:

Inductively coupled plasma emission spectrometer -Jobin-Yvon 1 m JY48P autosampler, DEC computer, & PET microcomputer interface to LIS (Remarks)

Calibration Range: 0 - 2 mg/l

Resolution: Four significant figures (0.0001 mg/l)

Sensitivity: NA

Instrument Detection Limit: .0142 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-.2 $\mu\text{g/g}$ to 200 $\mu\text{g/g}$

Accuracy- EPA #3 : 90 %

Precision of Controls-

mean	140 $\mu\text{g/g}$
std. dev.	11 $\mu\text{g/g}$
R.S.D.	7.8 %

B

Precision of Duplicates-low range	mid range	high range
s.d.	3.0	3.1
mean	22	63
		140

W 1 $\mu\text{g/g}$

T 5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	120	110
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QCV85-1 Upper limit	160	170
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REMARKS:- % extracted using orchard leaves (NBS-1571)

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN VEGETATION

Operating Range = 0.2000 to 200.0 ug/g

IN - RUN DUPLICATES

Range	<0.2000	0.2000 to 40.00	40.00 to 100.0	100.00 to 200.0	>200.0
no.	5	51	42	19	12
s.w.		2.9529	3.1326	10.6466	
mean		22.0928	62.6509	142.7485	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCV85-1	226	142.355	11.0319	7.75

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
rb	259	5.838	15.336

DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ZINC TEST CODE: ZNUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 533BA0 (AAS)
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried and ground to <1mm in a Wiley mill.

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 86
Procedure- Weigh 0.500 g of sample into a Vycor crucible and muffle at 150C for 15 min., 250C for 1 hr., and 500C for 3 hrs. Cool, wet the ash with DDW, followed by 3 ml HCl and 1 ml HNO₃. Digest contents, while covered with a watchglass, without boiling, for 4 hrs. Wash down the watchglass with DDW INTO the crucible. Add 2 drops of H₂O₂ and reduce the contents to 1 ml on a hot plate. Transfer digestate to a 15 ml centrifuge tube, add 0.2 ml of 5 % KCl and dilute to 10 ml with DDW. Mix, allow suspended particulates to settle out, and analyze by atomic absorption spectrophotometry (AAS) if less than five elements are requested and/or Na and Ag.

INTERFERENCES: Several, which may be compensated for by background correction.

REPORTING RESULTS: Two significant figures. ($\mu\text{g/g}$ Zn)

INSTRUMENTATION: For the analysis < 5 elements, Ag and Na.

A PE 5000 AAS, PE Auto-Sampler 50, and a PE automatic burner control interfaced with a Commodore Pet 4032, Tractor Printer 4022P & drives.

Calibration Range: 0 - 5 mg/l

Resolution: 0.01 mg/l

Sensitivity: Conc. for absorbance of approx. 0.2 units: 1.0 mg/l

Instrument Detection Limit: 0.02 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.40 $\mu\text{g/g}$ to 100 $\mu\text{g/g}$

Accuracy- ND

Precision of Controls-

	A	B
mean	160 $\mu\text{g/g}$	
std. dev.	10 $\mu\text{g/g}$	
R.S.D.	6.4 %	

Precision of Duplicates-low range	mid range	high range
s.d.	0.71	8.5
mean	28	70

W 0.5 $\mu\text{g/g}$

T 2.5 $\mu\text{g/g}$

CONTROL LIMITS: $\mu\text{g/g}$ Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit	140	130
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QCV85-1 Upper limit	180	190
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REMARKS:- % extracted using orchard leaves (NBS-1571).

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC-AA IN VEGETATION

Operating Range = 0.4000 to 100.0 mg/L

IN - RUN DUPLICATES

Range	<0.4000	0.4000 to 20.00	20.00 to 50.00	50.00 to 100.0	>100.0
no.	0	0	4	1	1
s.w.		0.0000	0.7070	8.4850	
mean		0.0000	29.7500	70.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
V85-1	10	162.000	10.3280	6.38

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/03/31

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Chlorine TEST CODE: CLUT CLWT SAMPLE TYPE: Vegetation
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L Pastorek

METHOD CODE: 009AX2 TYPE: X-ray fluorescence
REVISION NO: 84-01 DATE: August, 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g.
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- Pelletized samples must be stored in dessicator
Other- Air dried, and ground to less than 1 mm in a Wiley Mill.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure- Transfer 2.00 g sample to a plastic vial to which 0.50 g Hoeschst "C" wax is added.
Mix thoroughly and transfer to briquetting die. Press at 20 tons/sq. in. for 60 - 90 secs.
Analyze using a Siemens SRS-1 X-ray fluorescence spectrometer with following parameters: kV - 45; mA - 30; base line - 1.3; width - 2.5; vacuum - on; collimator - 0.4°; crystal PET; angle 65.340; detector - flow counter.
Intensity is determined from the average of two 4 sec. counts.
Concentration is determined from stored calibration data.

INTERFERENCES: Analysis protocol corrects for matrix effects due to Si, K, Ca, P, S.

REPORTING RESULTS: Cl as per cent to two decimal places

INSTRUMENTATION: Siemens SRS-1 X-ray fluorescence spectrometer with HP9825 and Commodore Pet computers; also briquetting die, Sartorius 4-place balance, Spex hydraulic press, Herzog hydraulic press.

Calibration Range: 0.006% to 1.5%

Resolution:

Sensitivity:

Instrument Detection Limit: 0.01%

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.002 to 1.0%

Accuracy- 100% at 0.075% level

Precision of Controls-

	A	B
mean	0.080	1.23
std. dev.	0.003	0.026
R.S.D.	3.8 %	2.1 %

Precision of Duplicates-	low range	mid range	high range
s.d.	0.004	0.011	0.022
mean	0.06	0.330	0.710

W .002 %

T .010 %

CONTROL LIMITS:

REMARKS:

- Samples of 1 g can be prepared when there is insufficient material for a 2 g pellet (usually true for mosses).
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CHLORINE IN VEGETATION

Operating Range = 0.0100to 1.0 %

IN - RUN DUPLICATES

Range	<0.0100	0.0100to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	51	87	30	27	23
s.w.		0.0040	0.0110	0.0220	
mean		0.0600	0.3300	0.7100	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CON A	60	0.080	0.0030	3.75
CON B	60	1.230	0.0260	2.11

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/09/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: FLUORIDE TEST CODE: FFUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 550BPO TYPE: Ion selective electrode
REVISION NO: Original, #1 DATE: 1983, Sept. 1987
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- None
Other- Samples are air-dried; and ground to <1mm in a Wiley Mill

SAMPLE PREPARATION: Partial Extn.-Yes Total Extn.- % Extracted- 95
Procedure- Weigh 0.500 g of sample into a screw capped plastic tube calibrated at 50 ml. Mix with 25 ml of 0.1N perchloric acid (HClO₄).

Transfer samples to a water bath and heat overnight or minimum of four hours at 80 ±5°C. Shake for one hour on shaker.
Dilute the contents to the 50 ml mark with 0.1N perchloric acid.
Analyze using a fluoride ion-selective electrode system, while stirring the suspension on a magnetic stirrer.

INTERFERENCES: High concentrations of Al (> 500 µg/g), Ca, Mg, and Fe
Hydrogen and hydroxyl ions (pH should be 5.5).
REPORTING RESULTS: Two significant figures. (µg/g F)
INSTRUMENTATION: Radiometer Ion 85 Ion Analyzer, fluoride selective electrode. PRS12 Alpha Printer, and SAC80 Sampler Changer.

Calibration Range: 0 - 1 µg/l
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 µg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 µg/g to 500 µg/g

Accuracy-ND

Precision of Controls-

	A	B
mean	141 µg/g	26 µg/g
std. dev.	9.7 µg/g	2.9 µg/g
R.S.D.	6.9%	11%

Precision of Duplicates-	low range	mid range	high range
s.d.	0.58	3.4	30
mean	12	150	270

W 0.5 µg/g T 2.5 µg/g

CONTROL LIMITS: µg/g Warning Limits (x±2σ) Rejection Limits (x±3σ)
Control Lower limit 120 20 110 17
Upper limit 160 32 117 35

REMARKS:

Control A = QCV85-1

Control B = Silver Maple

The samples are analyzed according to type and the appropriate controls analyzed with it.

SUMMARY REPORT OF QUALITY CONTROL DATA

FLUORIDE IN VEGETATION

Operating Range = 0.5000to 500.0 ug/g

IN - RUN DUPLICATES

Range	<0.5000	0.5000to100.00	100.00to250.0	250.00to500.0	>500.0
no.	23	125	7	2	0
s.w.		0.6280	3.3500	20.0000	
mean		13.4800	150.9000	273.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
vegqc	42	141.640	9.7180	6.86
redoak	28	1.610	0.4820	29.94
grass	33	13.140	1.3370	10.18
silvermapl	45	26.190	2.8900	11.03

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
blk	0	0.000	0.0000

DATE 87/04/23

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: NITROGEN -TOT TEST CODE: NNTKUR SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 595CC2 TYPE: Acid digestion/automated colourimetry
REVISION NO: Original, #1 DATE: 1970, Sept. 1987.
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - Approx. 20g
Container - Glass jars (4 oz.) with bakelite screw caps.
Preservative - None
Other - Samples are air-dried and ground to <1 mm in a Wiley Mill

SAMPLE PREPARATION: Partial Extn. - Total Extn. - Yes % Extracted -
Procedure - Digest a sample aliquot in 3 ml conc. H₂SO₄ for one hour until fuming occurs. Add 2 g of potassium persulphate and again take to fuming until the digestate clears. Titrate the digestate with 6.25N NaOH or 10 % H₂SO₄ to the methyl red end-point (pH 4.1) and make up to 100 ml. Analyze the sample by the automated phenate method.

INTERFERENCES:

REPORTING RESULTS: Two significant figures (mg/g N)
INSTRUMENTATION: Automated colourimetric system (Technicon or equivalent) with 630 nm filters.

Calibration Range: 0 - 10 mg/l NH₃ as N.

Resolution: 0.01 mg/l

Sensitivity:

Instrument Detection Limit: 0.05 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - 0.1 mg/g to 20 mg/g based on 0.05 g wt.

Accuracy - ND

Precision of Controls -

	A	B
mean	23 mg/g	
std. dev.	1.2 mg/g	
R.S.D.	5 %	

Precision of Duplicates -	low range	mid range	high range
s.d.	0.44	0.64	1.23
mean	5.4	14	26

W 0.2 mg/g

T 1.0 mg/g

CONTROL LIMITS: mg/g Warning Limits ($x \pm 2\sigma$) Rejection Limits ($x \pm 3\sigma$)

Control Lower limit 21 19

Upper limit 25 27

REMARKS: - % extracted using Orchard Leaves (NBS 1571).

- Total phosphorus (PPUT) may also be determined on the same digest.

SUMMARY REPORT OF QUALITY CONTROL DATA

NITROGEN IN VEGETATION

Operating Range = 0.1000 to 20.0 mg/g

IN - RUN DUPLICATES

Range	<0.1000	0.1000 to 4.00	4.00 to 10.00	10.00 to 20.0	>20.0
no.	0	9	35	76	19
s.w.		0.2080	0.4680	0.6570	
mean		3.0900	6.6500	14.4800	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCVB-2	63	22.960	1.1627	5.06

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/02/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: PHOSPHORUS-TOT TEST CODE: PPUT SAMPLE TYPE: TERREST. VEG
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 314CC2 TYPE: Acid digestion/automated colourimetry
REVISION NO: Original, #1 DATE: 1970, Sept. 1987
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g.
Container-glass glass jar (4 oz.) with bakelite screwcap
Preservative-
Other-samples are air-dried and ground to <1mm in a Wiley Mill

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-105%
Procedure-Digest 0.02 to 0.04g of sample in 3 ml conc. H₂SO₄ for one hour until fuming occurs. Add 2 g of potassium persulphate and again take to fuming until the digestate clears. Titrate digestate with 6.25N NaOH or 10% H₂SO₄ to the methyl red end point (pH 4.1) and make up to 100 ml. Analyze by the automated SnCl₂ reduced phosphomolybdate method.

INTERFERENCES:

REPORTING RESULTS: Two significant figures (mg/g P)
INSTRUMENTATION: Automated colourimetric system (Technicon or equivalent) with 660 nm filters.

Calibration Range: 0 - 2 mg/l PO₄ as P

Resolution: 0.002 mg/l

Sensitivity: ND

Instrument Detection Limit: 0.01 mg/l

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.02 - 4 (mg/g based on 0.05 g sample)

Accuracy-105 % - orchard leaves (NBS 1571)

Precision of Controls-

A
mean 2.2 mg/g
std. dev. 0.10 mg/g
R.S.D. 4 %

B

Precision of Duplicates-low range	mid range	high range
s.d. 0.07	0.06	0.19
mean 0.50	1.22	2.93

W 0.02 mg/g

T 0.10 mg/g

CONTROL LIMITS: ug/g Warning Limits ($\pm 2\sigma$) Rejection Limits ($\pm 3\sigma$)

Control Lower limit 2.0 1.9

Upper limit 2.4 2.5

REMARKS:- "% extracted" determined using NBS 1571 (orchard leaves)
- Total nitrogen (NNTKUR) may also be determined on same sample digest

SUMMARY REPORT OF QUALITY CONTROL DATA

PHOSPHORUS IN VEGETATION

Operating Range = 0.0200 to 4.0 mg/g

IN - RUN DUPLICATES

Range	<0.0200	0.0200 to 0.80	0.80 to 2.00	2.00 to 4.0	>4.0
no.	0	51	62	20	6
s.w.		0.0700	0.0570	0.1900	
mean		0.5000	1.2200	2.9300	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
QCVB-2	64	2.178	0.0975	4.48

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/02/02

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Sulphur TEST CODE: SSUT SSWT SAMPLE TYPE: Vegetation
UNIT: Vegetation/Soil/Sediment SUPERVISOR: L. Pastorek

METHOD CODE: 009AX2 TYPE: X-ray fluorescence
REVISION NO: 84-01 DATE: August, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approximately 20 g.
Container- Glass jar (4 oz) with bakelite screw cap
Preservative- Pelletized samples must be stored in dessicator
Other- Air dried, and ground to less than 1 mm in a Wiley Mill.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure- Transfer 2.00 g sample to a plastic vial to which 0.50 g Hoeschst "C" wax is added.
Mix thoroughly and transfer to briquetting die. Press at 20 tons/sq. in. for 60 - 90 secs for smooth, durable, homog. pellet.
Analyze using a Siemens SRS-1 X-ray fluorescence spectrometer with following parameters: kV - 45; mA - 30; base line - 1.3; width - 2.5; vacuum - on; collimator - 0.4°; crystal - graphite; angle - 106.330.
Intensity is determined from the average of two 4 sec. counts.
Concentration is determined from stored calibration data.

INTERFERENCES: Analysis protocol corrects for matrix effects due to Si, Cl, Ca, P, K.

REPORTING RESULTS: S as per cent to two decimal places

INSTRUMENTATION: Siemens SRS-1 X-ray fluorescence spectrometer with HP9825 and Commodore Pet computers; also briquetting die, Sartorius 4-place balance, Spex hydraulic press, Herzog hydraulic press.

Calibration Range: 0.02% to 4.00%

Resolution:

Sensitivity:

Instrument Detection Limit: 0.02%

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.010% to 0.50%

Accuracy- 101% at 0.179% level

Precision of Controls-

	A	B
mean	0.180	0.650
std. dev.	0.005	0.014
R.S.D.	2.8 %	2.2 %

Precision of Duplicates-	low range	mid range	high range
s.d.	0.0050	0.0080	0.0110
mean	0.060	0.160	0.34

W .002 %

T .010 %

CONTROL LIMITS:

REMARKS:

- Samples of 1 g can be prepared when there is insufficient material for a 2 g pellet (usually true for mosses).
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

SULPHUR

IN VEGETATION

Operating Range = 0.0100 to 0.5 %

IN - RUN DUPLICATES

Range	<0.0100	0.0100 to 0.10	0.10 to 0.25	0.25 to 0.5	>0.5
no.	1	46	128	36	7
s.w.		0.0050	0.0080	0.0110	
mean		0.0600	0.1600	0.3400	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CON A	60	0.180	0.0050	2.78
CON B	60	0.650	0.0140	2.15

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
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DATE 87/09/11



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